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by Tomoko Sano, James Catalano, Daniel Casem, and Dattatraya Dandekar

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Weapons and Materials Research Directorate, ARL

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MICROSTRUCTURAL AND MECHANICAL BEHAVIOR CHARACTERIZATION OF ULTRASONICALLY CONSOLIDATED TITANIUM-ALUMINUM LAMINATES

Tomoko Sano¹, James Catalano¹, Daniel Casem², and Dattatraya Dandekar²

¹U.S. Army Research Laboratory
Attn: AMSRD-ARL-WM-MD; Aberdeen Proving Ground, MD, 21005, USA

²U.S. Army Research Laboratory
Attn: AMSRD-ARL-WM-TD; Aberdeen Proving Ground, MD, 21005, USA

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Abstract

Multilayered hybrid metal laminates have been studied for structural applications due to their potential for higher strength, toughness, and stiffness. The goal of this study was to modify the microstructure and mechanical properties of commercial purity titanium (CP-Ti) and 1100 aluminum (Al) laminates for potential applications in mine blast mitigation. Alternating layers of 50 μm thick CP-Ti and Al layers were ultrasonically consolidated. To provide high hardness and stiffness, the consolidated laminates were heat-treated in a variety of conditions to form intermetallic titanium aluminide (TiAl_3) layers. The resulting CP-Ti/ TiAl_3 /Al laminates were characterized by scanning electron microscopy. Plate impact testing, an instrumented laboratory scale test to characterize the dynamic spall behavior of the material, was conducted on select laminates. Based on these results, the CP-Ti/ TiAl_3 /Al laminate had a higher spall strength compared to the CP-Ti/Al laminate.

Introduction

Titanium alloys and intermetallics have been used in light weight structural and armor applications. The TiAl_3 intermetallic compound, like most intermetallics, has high hardness and stiffness, but is brittle [1]. However when combined in a metal-intermetallic laminate system with Ti and Al layers, the brittle property is offset by the strength and ductility of Ti and Al, increasing the toughness. In this study, Ti- TiAl_3 -Al metal-intermetallic laminates were ultrasonically consolidated, processed, and characterized for microstructure and spall strength.

Ultrasonic consolidation is a high speed process that uses ultrasonic oscillation to produce friction leading to adhesion of dissimilar metallic foil layers. The ultrasonic weld, or bond, is formed between the metal foils in less than 250 milliseconds, by localized slip that breaks up the surface oxide and causes elastoplastic deformation at the interface. The sonotrode, which produces the ultrasonic oscillation while rotating over the metal surface, applies low pressures below 50 MPa, and does not produce heat. These aspects make it ideal for fragile structures and heat sensitive metals to be consolidated. In addition, sensors can be embedded between the metallic foils for measurements of stimuli or for adaptive response.

Experimental Procedure

Laminate Sample Processing

Alternating layers of 50 μm thick CP-Ti and Al were ultrasonically consolidated (UC) by Solidica Inc. Repeated sets of an Al and a CP-Ti double layer were laid down over a 1.27 cm thick base 3003 Al, each double layer ultrasonically welded by the sonotrode oscillating over the Ti surface, to form 0.64 cm thick laminates of CP-Ti/Al on the base Al. A scanning electron microscopy (SEM) image of a cross section of the as-received laminate is shown in Fig. 1. The laminates were then cut into roughly 5.0 cm by 5.0 cm by 1.9 cm samples and heat treated.

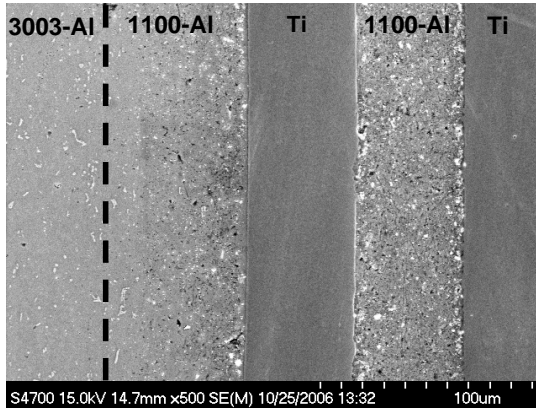


Figure 1. Scanning electron microscopy image of a cross section of the as received unreacted laminate.

Following the studies by Xu et al [2] to form TiAl_3 , the annealing temperature of 550°C , which is below the melting point of Al, was chosen [3]. Six different heat treatments at 550°C were conducted in vacuum or in air, in the hot press with or without pressure. Table I shows the different heat treatments conducted.

Table I. Heat Treatments Conducted

Experiment	1	2	3	4	5	6
Time at 550°C (h)	1	1	1	1	3	12
Pressure (MPa)	6	6	0	0	6	Minimal
Environment	Vacuum	Air	Vacuum	Air	Vacuum	Vacuum

After the heat treatments, smaller samples were sectioned off with Struers Discotom abrasive cutoff saw, then encased in Bakelite mount and polished. Each sample was polished with 240 grit, 320 grit, 400 grit, 600 grit, and 800 grit SiC paper, then polished on the vibramet with 5 μm alumina slurry for 1 h, 1 μm alumina slurry for 0.5 h, and finished with a 0.06 μm colloidal silica for 0.5 h.

Spall Strength Test

The procedure to determine spall strength is to impact a plate of the sample material with a flyer plate of known properties at normal incidence. As a result of impact, a compressive shock wave traverses the target and reflects from the free-surface, in tension, back into the shocked material. This reflection then interacts at some point with the release wave that follows the original shock. This produces tension which can, if it is of sufficient duration and magnitude, spall the material. The particle velocity at the target free-surface is monitored with laser interferometry (VISAR, [4]). The specimens were oriented such that the spall plane is parallel to the individual laminates.

Since the spall strength of the laminate was expected to be limited by the strength of the Al even with perfect bonding, the spall strength of the solid Al, both heat-treated and untreated were tested. Laminates of heat treated and as received UC Al were also tested to determine the bonding quality of the laminates. For the UC CP-Ti/Al laminates, as received samples and heat treated laminates (using the Experiment 1 heat treatment parameters) were tested.

Results and Discussion

Microstructure

X-ray diffraction (XRD) of the heat treated UC laminates indicated the Ti reacted with the Al to form the TiAl_3 intermetallic phase. This is in agreement with previous researchers who observed TiAl_3 formation between the Ti and Al layers after heat treatment [2, 3, 5-7]. The XRD pattern of the sample from experiment 1, with labeled Ti, Al, and TiAl_3 peaks, is shown in Fig. 2.

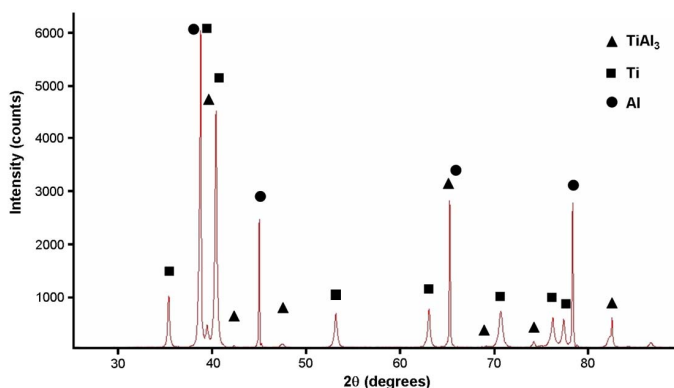


Figure 2. XRD Pattern of a sample hot pressed at 550°C with 6 MPa of pressure for 1 h in vacuum.

The samples that were hot pressed with 6 MPa of pressure, regardless of the environment, exhibited more distinct TiAl_3 layer interfaces between the CP-Ti and Al layers. Experiment 1 and 2 did not show any cracking of the TiAl_3 layer. Experiment 5 showed cracking only at the thick TiAl_3 layers. In addition, the pressure appeared to have assisted in the reaction of Ti with Al, forming a homogenous TiAl_3 layer. In contrast, the samples heat treated without any applied pressure (experiment 3 and 4) exhibited cracking in the TiAl_3 phase, perpendicular to the length

of the layer. A back scattered electron micrograph obtained by a SEM of the polished cross sectioned surface of the sample from experiment 3 shows this cracking in Fig. 3. In addition, some parts of the TiAl_3 layers were less homogenous.

Comparing the results between experiment 1 and 2, as well as results between 3 and 4, the furnace environment had an effect on the Ti-Al reaction. Regardless of the pressure condition, thicker TiAl_3 layers were observed when heat treated in vacuum rather than in air. However more experiments are necessary to determine the difference in the reaction rates.

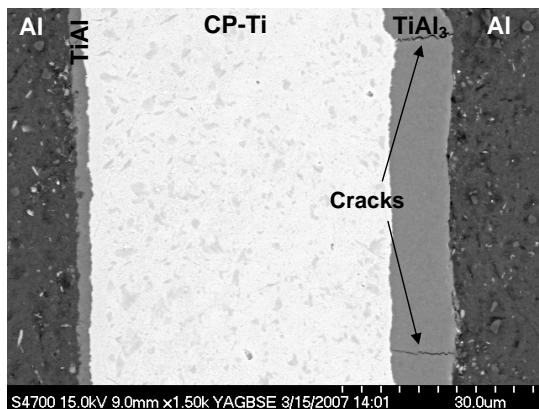


Figure 3. Back scattered diffraction micrograph of the reacted laminate layers. Cracks were observed in the thicker TiAl_3 layers.

For all heat treatments, the TiAl_3 layer on one side of Al was less wide than the other side. As shown in Fig. 3, the right side of the CP-Ti has a thicker TiAl_3 layer than the left side of the Al. This was attributed to the sonotrode causing more surface roughening and breaking up the oxide layer on one surface of the CP-Ti; the right Ti surface as shown in Fig. 3. The rougher surface, having a larger surface area with less oxide, had more reaction between Ti and Al, resulting in a thicker TiAl_3 layer. Experiment 6, which unintentionally cycled minimal pressure throughout the heat treatment, delaminated after the heat treatment and could not be examined. The heat treatment properties of the experiments and the resulting TiAl_3 layer are shown in Table II.

Table II. Heat Treatment and Resulting TiAl_3 Layer

Experiment	1	2	3	4	5	6
Time at 550°C (h)	1	1	1	1	3	12
Pressure (MPa)	6	6	0	0	6	Minimal
Environment	Vacuum	Air	Vacuum	Air	Vacuum	Vacuum
TiAl_3 Thickness (μm)	1 - 5	0.5 - 1	2 - 10	< 0.5	3 - 30	-
TiAl_3 Cracked?	No	No	Yes	Yes	Yes	Delaminated

Spall Strength

The spall strength can be related to the size of the so-called “pull-back” signal seen in the velocity trace following the initial compressive wave, as described by the following:

$$\text{Spall Strength} = \frac{1}{2} \cdot \rho \cdot (\text{shock speed}) \cdot (\text{pull-back velocity}) \tag{1}$$

where ρ is the density of the material. Due to the uncertainty of the material properties in the present experiments, we have elected to report the spall strength in terms of the more qualitative measure of the pull-back velocity, following the linear-elastic material spall strength equation (Eq. 1) above, rather than an actual strength. The pull-back velocities, along with other relevant conditions of each test, are given in Table III.

Table III. Spall Test Parameters and Results

Flyer			Target			Impact Speed (m/s)	Pull-back Velocity (m/s)
Material	Thickness (mm)	Heat Treatment	Material	Thickness (mm)	Heat Treatment		
Solid Al	2.04	None	Solid Al	3.84	None	200	118
Solid Al	2.00	None	Solid Al	4.01	None	204	115
Solid Al	2.02	None	Solid Al	3.89	3 hours	210	130
Solid Al	1.96	None	UC Al	3.97	None	204	22
Solid Al	2.04	None	UC Al	4.06	None	200	20
Solid Al	2.03	None	UC Al	3.82	3 hours	210	38
Solid Al	1.96	None	UC TiAl	3.98	None	235	5
Solid CP Ti	1.99	None	UC TiAl	3.98	None	235	4
Solid Al	1.98	None	UC TiAl	3.94	1 hour	228	45

From this data, four observations can be made. First, when the solid Al is heat-treated, there is a small gain in spall strength (~12%). When the UC Al is heat-treated, there is a moderate gain in spall strength (~81%). However, when the CP-Ti/Al laminate is heat-treated, there is a much larger gain in spall strength (~1000%), indicating that the formation of TiAl₃ greatly increases the bond strength. It should be note that the pull-back velocity of the heat-treated Cp-Ti/Al is about 1/3 that of the heat-treated solid Al, which indicates that the spall plane is occurring at the bond, and not within an Al layer.

Conclusions

Alternating CP-Ti and Al layers were ultrasonically consolidated into laminates and heat treated to form intermetallic layers, which were determined by XRD to be TiAl₃. SEM analysis of the post-heat treated samples showed the heat treatment parameters of pressure and environment had an effect on the reaction between Ti and Al. On the CP-Ti surfaces made rougher due to the sonotrode, consistently thicker TiAl₃ was observed.

The spall strength tests were conducted by plate impact on solid AL, UC Al laminates, and UC CP-Ti/Al laminates. Heat treatment was observed to increase the spall strength, however for the Cp-Ti/Al laminate, the increase in spall strength was most likely due to the increase in bond strength with the formation of TiAl₃ layers.

Though the kinetics of the Ti-Al reaction and the effect of TiAl₃ layer thickness on spall strength need to be further explored, these initial results show heat treating UC CP-Ti/Al

laminates forms TiAl_3 intermetallic layers which not only increases the bond between the CP-Ti and Al layers, but also increases the strength and of the laminate material.

References

1. D.R. Bloyer, K.T. Venkateswara Rao, and R.O. Ritchie, "Resistance-Curve Toughening in Ductile/Brittle Layered Structures: Behavior in Nb/Nb₃Al Laminates," *Mater. Sci. Eng. A*, 216 (1996), 80-90.
2. L. Xu, Y.Y. Cui, Y.L. Hao, and R. Yang, "Growth of Intermetallic Layer in Multi-Laminated Ti/Al Diffusion Couples," *Mater.Sci. Eng. A*, 435-436 (2006) 683-47.
3. Y. Mishin and C. Heraig, "Diffusion in the Ti-Al System," *Acta Mater.*, 48 (2000) 589-623.
4. L.M. Barker and R.E. Hollenback, "Laser Interferometer for Measuring High Velocities of Any Reflecting Surface," *J. Appl. Phys.*, 43 (11) (1972) 4669-4675.
5. L.M. Peng, H. Li, and J.H. Wang, "Processing and Mechanical Behavior of Laminated Titanium-Titanium Tri-Aluminide ($\text{Ti-Al}_3\text{Ti}$) Composites," *Mater.Sci. Eng. A*, 406 (2005) 309-18.
6. D.J. Harach and K.S. Vecchio, "Microstructure Evolution in Metal-Intermetallic Laminate (MIL) Composites Synthesized by Reactive Foil Sintering in Air," *Metall. Mater. Trans.*, 32A (6) (2001) 1493-1505.
7. R. Jiangwei, L. Yajiang, and F. Tao, "Microstructure Characteristics in the Interface Zone of Ti/Al Diffusion Bonding," *Mater. Letters*, 56 (2002) 647-52.

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